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CERTIFICATE

This certificate is issued in support of an application for Patent registration in a country outside New Zealand pursuant to the Patents Act 1953 and the Regulations thereunder.

I hereby certify that annexed is a true copy of the Provisional Specification as filed on 10 December 2002 with an application for Letters Patent number 523100 made by RICHARD HAMILTON ARCHER;DEREK ROBIN HAISMAN.

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NEW ZEALAND PATENTS ACT 1953

PROVISIONAL SPECIFICATION

A PROCESS FOR PRODUCING A CARBOHYDRATE COMPOSTION

We, RICHARD HAMILTON ARCHER, New Zealand citizen of 5 Hillgrove Place, Palmerston North, New Zealand; and DEREK ROBIN HAISMAN, a British citizen of 20 Lees Road, RD5, Fielding, New Zealand, do hereby declare this invention to be described in the following statement:

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A PROCESS FOR PRODUCING A CARBOHYDRATE COMPOSITION

FIELD OF THE INVENTION

The present invention relates to a process for the production of a carbohydrate composition comprising a mixture of sugars specifically, although by no means exclusively as a syrup, from a starting material of lactose. The present invention also relates to the compositions produced by the process of the invention as well as the foods and drinks containing the compositions.

BACKGROUND

Carbohydrate compositions comprising a mixture of sugars, such as lactose, glucose, galactose, fructose etc are useful as food and drink additives in commercial food and drink production. For example, compositions comprising approximately 40-50% galactose, 25-30% fructose and 25-30% glucose are useful in the manufacture of sports drinks and energy snacks for sportsmen, confectionery, or for people having special food requirements such as diabetics (EP 0499165).

Known processes for producing such a composition include simple admixing of individual purified sugars in the required amount. However, sugars in their pure form can not only be quite expensive but also the purity and therefore quality for each sugar may vary from source to source and therefore result in variability of the end composition.

Other known processes include one or more enzyme conversions of one sugar to another thereby producing a mixture of at least two sugars. Additional sugars may then added from a purified source to complete the desired composition.

For example US 3852496 describes a method of producing a sweetening composition from whey containing lactose using immobilised beta-galactosidase (lactase) and glucose isomerase. The lactose is passed over a flow-through column containing immobilised lactase to produce glucose, galactose and unhydrolysed lactose. This composition is either used directly or treated with glucose isomerase to produce a composition containing fructose, glucose, galactose and lactose.

Poutanen et al (1978) describes the conversion of glucose to fructose in hydrolysed whey and lactose syrups by glucose isomerase treatment using immobilised enzyme

technology. To increase efficiency of the process, a purified source of glucose was added to the hydrolysed lactose syrup before isomerisation to increase the relative content of fructose and therefore to increase the sweetness of the resulting composition.

Chiu and Koskowaski (1985) describe the hydrolysis of whey lactose followed by glucose isomerisation with added glucose and subsequent purification of fructose syrup.

Harju and Kruela (1980) describe the hydrolysis of whey lactose to produce a mixture of sugars which increases in sweetness to a maximum when hydrolysis is 80% complete. Further hydrolysis above this level does not increase the sweetness but does significantly increase the cost of hydrolysis. To increase sweetness further, glucose is isomerised to fructose.

The above prior art methods are mainly concerned with obtaining a carbohydrate compositions having maximum sweetness. Galactose is a carbohydrate which is not particularly sweet and not, therefore a desirable component of those prior art compositions.

Galactose is a particularly desirable ingredient of compositions which are useful in sports drinks etc (US 5780094) as it is easily and quickly absorbed to provide a rapid energy source as well as aiding in replenishment of glycogen reserves in the liver. Unfortunately, at present, it is not possible to simply add pure galactose to the prior art compositions as sources of galactose are not available in sufficient commercial quantities for large scale consumer products. In addition, even if sufficient quantities were available, such galactose would be prohibitively expensive and could not compete with conventional cheaper energy sources used in commercial sports drinks such as sucrose. This is because it is difficult to separate galactose from other sugars with which it occurs naturally, such as glucose, arabinose, mannose, fructose etc. The most common source of galactose is in milk or in pectin where it occurs as a side chain, and requires a complex separation process.

It is an object of the present invention to provide a process for producing a composition comprising a mixture of sugars including galactose and/or to provide a cheap and convenient method of producing purified galactose which overcomes, at least to some extent, the problems aforesaid and/or provides the public with a useful choice.

SUMMARY OF THE INVENTION

The present invention provides a process for the production of a composition comprising a mixture of approximately 10-50% galactose, 0-48% glucose, 1-25% fructose, 1-48% gluconic acid and 0-25% "others" comprising unconverted lactose and non-lactose di- and oligo- saccharides as a % of the total carbohydrate present. Preferably the composition comprises 30-50% galactose, 10-40% glucose, 5-25% fructose, 1-15% gluconic acid and 1-10% "others". Most preferably, the composition comprises 45% galactose, 25% glucose, 20% fructose, 5% gluconic acid and 5% "others".

In a first embodiment, the invention provides a process comprising the steps:

(i) hydrolysis of lactose to produce glucose and galactose;

(ii) partial isomeristion of the glucose to fructose; and

(iii) partial oxidation of the glucose to gluconic acid;

to produce a composition comprising a mixture of galactose, glucose, fructose, gluconic acid and unconverted lactose and non-lactose di- and oligo- saccharides without the need of any purification steps.

The process may be carried out as a continuous, semi continuous, batch, sequence batch or single pot process.

The isomerisation step (ii) may be carried out either before or after the oxidation step (iii).

The hydrolysis step (i) and oxidation step (iii) may be carried out simultaneously.

Alternatively, the product of step (i) may be separated into three streams and the first stream not treated further and the second and third streams treated according to steps (ii) or (iii) respectively and the products of each stream combined to provide a final composition according to the invention.

In a second embodiment, the invention provides a composition produced by the process, wherein said composition comprises a mixture of galactose, glucose, fructose, gluconic acid and unconverted lactose and non-lactose di- and oligo-saccharides. The undiluted composition is generally in the form of a syrup of 40 to 80° Brix but this may be diluted to any desired strength.

The composition comprises approximately 10-50% galactose, 0-48% glucose, 1-25% fructose, 1-48% gluconic acid and 0-25% "others" comprising unconverted lactose and non-lactose di- and oligo- saccharides. Preferably the composition comprises 30-50% galactose, 10-40% glucose, 5-25% fructose, 1-15% gluconic acid and 1-10% "others". Most preferably, the composition comprises 45% galactose, 25% glucose, 20% fructose, 5% gluconic acid and 5% "others".

In a third embodiment, the invention provides a food or drink containing the composition of the invention, and particularly a sports energy bar or sports drink.

In a fourth embodiment, the present invention provides a process for the production of galactose comprising the steps

(i) hydrolysis of lactose to produce glucose and galactose;

(ii) partial isomeristion of the glucose to fructose; and

(iii) optional partial oxidation of the glucose to gluconic acid;

(iv) crystallization of galactose; and

(v) recovery of pure galactose crystals from the mother liquor.

In a fifth embodiment, the present invention provides pure galactose produced by the process of the invention.

In a sixth embodiment, the present invention provides a composition comprising the mother liquor produced by the process of the invention and its use as a sweetener in the food industry, and in particular, in the dairy food industry.

The invention will now be described by reference to the figure of the accompanying drawing in which:

Figure 1 shows a schematic diagram of the process of the present invention.

DETAILED DESCRIPTION

The present invention is concerned with a process for the production of a composition comprising a mixture of galactose, glucose, fructose, gluconic acid and unconverted lactose and non-lactose di- and oligo- saccharides, from lactose as a starting material. Such compositions are particularly useful in the preparation of sports drinks and sports bars as a source of readily absorbable energy after exercise. Galactose is especially useful in this regard and the present invention is also concerned with a process for the production of galactose.

In a first embodiment the present invention provides a process comprising the steps:

(i) hydrolysis of lactose to produce glucose and galactose;

(ii) partial isomeristion of the glucose to fructose; and

(iii) partial oxidation of the glucose to gluconic acid;

to produce a composition comprising a mixture of galactose, glucose, fructose, gluconic acid and unconverted lactose and non-lactose di- and oligo- saccharides without the need of any purification steps. This process is shown schematically in Figure 1.

The process may be carried out as a continuous, semi-continuous, batch, sequenced batch or single pot process.

The isomerisation step (ii) may be carried out either before or after the oxidation step (iii).

The hydrolysis step (i) and oxidation step (iii) may be carried out simultaneously.

Alternatively, the product of step (i) may be separated into three streams and the first stream not treated further and the second and third streams treated according to steps

(ii) or (iii) respectively and the products of each stream combined to provide a final composition according to the invention.

Alternatively, the product of the partial isomerisation step (ii) may be split and a portion subjected to partial oxidation (step (iii)) and the remainder combined with the product of the partial oxidation step to produce a composition of the invention.

Alternatively, the product of the partial oxidation step (iii) may be split and a portion subjected to partial isomerisation (step (ii)) and the remainder combined with the product of the partial isomerisation step to produce a composition of the invention.

Preferably, the process comprises hydrolysis step (i) followed by partial oxidation step (iii) wherein the majority of this stream (eg 85%) is further processed via partial isomerisation step (ii) and the remaining portion of this stream (by pass) is combined with the product of the step (ii) to produce a composition of the invention having a desired fructose content..

The lactose source may be selected from the group comprising milk; UF permeate derived from whole milk, skim milk, whey or milk serum; pure lactose; whey; deproteinated whey; demineralised whey; decalcified whey; UF permeate derived from deproteinised, demineralised or decalcified whey; or any combination thereof.

The hydrolysis step (i) may be achieved chemically, including the use of mineral acids, strong cation exchange resins, enzymatically using one or more hydrolytic enzymes or in a bioreactor.

The mineral acids may comprise a weak solution (0.01 – 0.1% of total weight of lactose) of strong mineral acids such as hydrochloric acid, suphuric acid, phosphoric acid or nitric acid.

The hydrolytic enzyme (beta-galactosidase, also known an lactase) may be free or immobilised and may be sourced from *Kluyveromyces lactis, Kluyveromyces fragilis, Kluyveromyces marxianus, Saccharomyces fragilis, Streptococcus thermophilus, Aspergillus oryzae, Aspergillus niger, Lactobacillus bulgaricus, Lactobacillus helviticus, Lactobacillus salivarius, Lactobacillus fermentum, Lactobacillus casei, Lactobacillus acidophilus, Steptococcus lactis, Bifidobacterium bifidum, Bifidobacterium longum, Bifidobacterium adolescentis, Bifidobacterium breve, Bacillus subtilis, Escherichia coli, Sulfolobus sp, especially Sulfolobus solfatarius,*

Pyrococcus fusiosus, green coffee beans, jack beans, bovine liver, and bovine testes and any other suitable source either alone or in combination.

The pH of the hydrolysis reaction mixture is maintained at pH 6.8 – 7.5, preferably 7.1-7.3, most preferably 7.2 by using a strong acid or alkali as required (e.g. NaOH, KOH, HCl, KH_2PO_4, K_2HPO_4, potassium or sodium citrate, magnesium carbonate, sulphuric acid, citric acid or a mixture thereof) under suitable conditions according to the source of the enzyme, its activity, temperature and amount of starting material as would be understood by a skilled person and as set out in the manufacturers instructions. General conditions are approximately 40-50°C, preferably 45°C for approximately 8 hours.

The isomerisation step (ii) may be achieved chemically or enzymatically. When an enzyme is used, such a glucose isomerase enzyme may be free or immobilized and may be sourced from *Actinoplanes missiourensis, Bacillus coagulans, Streptomyces murinus, Escherichia coli and Arthrobacter species.* Again the reaction conditions are dependant on the source of the enzyme and manufacturers' recommendations may be followed. Generally, preferred conditions are similar to those used in the industrial production of high fructose corn syrup where starch derived dextrose is converted to a fructose/dextrose mixture. For the present invention, general conditions are 55-62°C and 0.5-5 bed volumes/hour.

This step may be carried out in a membrane bioreactor. Preferably, this step carried out using an immobilised enzyme in a column format.

It is desirable for sports drinks to have a relatively low glycemic index and the presence of sugars other than glucose, or sugars which may be converted to glucose in the small intestine, is therefore important when formulating such drinks. Galactose, for example, acts to reduce the glycemic index in a sports drink. The oxidation step (iii), however, serves to further reduce the glycemic index by converting some of the glucose present to gluconic acid.

The oxidation step (iii) may be achieved chemically or enzymatically. The enzyme conversion process requires two enzymes, a glucose oxidase and a catalase. The catalase may be a contaminant of the oxidase enzyme or may be commercially produced as a single product. Alternatively, both enzymes may be added separately to the reaction mixture. Such enzymes may be free or immobilized. The oxidase enzyme may be sourced from *Penicillium notatum, Penicillium glaucanum, Penicillium*

amagosakiense and *Aspergillus niger*. The catalase enzyme may be sourced from *Aspergillus niger, Penecillium* species (as for oxidase, above) and *Micrococcus lysodeikticus*. The reaction conditions are dependent upon the source of the enzyme, its activity, amount of reactant etc and the manufacturers instructions may be followed. Generally, the reactions take place at 45-60°C, preferably 55-58°C for 2-4 hours whilst in contact with air/oxygen. The pH of the reaction mixture is maintained around 4.5-6.5, preferably 5.6 by adding base. Alternatively, the oxidation step (iii) may be carried out in a membrane bioreactor. This step may also be carried out under hyperbaric pressure conditions as described in US 4,345,031.

The oxidation step converts some of the glucose present in the reaction mixture to gluconic acid. Gluconic acid is considered to be a particularly desirable component of the composition of the present invention for several reasons. Firstly, by reducing the amount of glucose present in the composition, as discussed above, the glycemic index of the composition is reduced. Secondly, the acidity of the gluconic acid is desirable for sports drinks' applications and thirdly, the gluconic acid present acts to improve the flavour of the composition and subsequently diluted sports drinks as it assists in disguising the sodium flavour.

The hydrolysis step (i) and oxidation step (iii) may be carried out simultaneously where conditions allow, for example, where the agent used to control pH is compatible with hydrolysis (such as KOH).

The process of the present invention may also include a number of optional filtration, ion exchange and carbon purification steps to purify the syrup produced by the process as would be appreciated by a skilled person. The process may also include pH adjustments to be made periodically to improve the overall efficiency of the process.

The composition produced by this process comprises approximately 10-50% galactose, 0-48% glucose, 1-25% fructose, 1-48% gluconic acid and 0-25% "others" comprising unconverted lactose and non-lactose di- and oligo- saccharides as a % of the final carbonate present. Preferably the composition comprises 30-50% galactose, 10-40% glucose, 5-25% fructose, 1-15% gluconic acid and 1-10% "others". Most preferably, the composition comprises 45% galactose, 25% glucose, 20% fructose, 5% gluconic acid and 5% "others".

The non-lactose di- and oligo- saccharides, together with the unconverted lactose ("others") make up approximately 5% of the total carbohydrate content of the

composition. This "other" component comprises bifidogenic material and may have a beneficial health effect in the sports drinks, sports bars and other food and drinks to which the composition is added. In addition, this 'other' component may provide some calorific value. Without being bound by theory, although it is not expected that these di- and oligo- saccharides will be adsorbed in the upper gastrointestinal tract, it is likely that they will be converted to short chain fatty acids and may be adsorbed in the colon to provide an energy source. It is also an advantage in the concentrated syrup of the invention in that this "other" component, particularly the non-lactose di- and oligo- saccharide component, acts to maintain all of the sugars in solution or inhibit crystallisation to some degree.

The composition produced by the process of the present invention is generally in the form of a syrup of approximately 5° Brix. This composition may be used directly in a sports drink without further dilution. However, preferably the composition produced by the process of the invention s in the form of a concentrated syrup of 40 to 80° Brix. The composition is concentrated by one or more evaporation steps. In particular, when step (i) is carried out alone or is combined with step (iii), the process may be carried out under dilute conditions, ie. >75%-95% water (or a total solids content of 5-25%) and a thermal evaporation step carried out before step (ii) to increase the total solids to 40-60%. The syrup may be further dried in a rotary evaporator, for example, if desired.

Preferably, the composition is in the form of a concentrated syrup and may be used as an additive in sports drinks and sports bars. In general 2.5-7.5% of the concentrated syrup is added to water and other ingredients such as flavours, to produce a sports drink. A major advantage of the process of the present invention is the flexibility of the process steps which may be varied to produce a final syrup of any desired composition.

One problem associated with the syrup of the invention is that it is prone to crystallization of the galactose component at temperatures between the range –10°C to +30°C depending on the concentration of the syrup. Therefore, to avoid crystallisation, the syrup must be kept at a temperature outside of this range. This is not a problem once the concentrated syrup has been diluted into a sports drink. As mentioned above, the presence of the galactooligosaccharides in the 'other' component of the composition is thought to act to inhibit cyrstallisation, but crystallisation of galactose in particular, may still occur outside the abovementioned temperature range.

However, as discussed above, a pure source of galactose is not readily available as a large volume item of commerce, and a further embodiment of the present invention provides a process for the production of galactose comprising the steps

(i) hydrolysis of lactose to produce glucose and galactose;

(ii) partial isomeristion of the glucose to fructose;

(iii) optional partial oxidation of the glucose to gluconic acid;

(iv) crystallization of galactose; by evaporation and/or cooling and

(v) recovery of pure galactose crystals from the mother liquor.

Steps (i), (ii) and (iii) of this process are the same as described above and may be carried out in the order and manner described above. Step (iv) may be carried out by cooling the syrup of the invention to a temperature between the range –10°C to +30°C, preferably 4-20°C, whereby crystallisation of pure galactose commences. Galactose crystallizes out of solution more efficiently at lower temperatures. Preferred conditions are 4°C for up to 48 hours. The crystals may then be recovered in step (v) by centrifugation or filtration and washing with ice cold water the pure galactose may be air dried using a fluid bed dryer. This process is effective at crystallizing approximately 50% of the galactose present in the syrup composition of the invention. For example, if the syrup contains 48% of the carbohydrate as galactose, approximately 24-32% of this will crystallise as pure galactose. This process may be used for small or large scale manufacture of pure galactose.

The efficiency of crystallisation is affected by the concentration of the syrup and temperature, as described above, and also by the complexity of the sugars present. The more complex carbohydrate present in the syrup, the more crystallisation is inhibited. In particular, the more "others" component present, the more crystallization is inhibited. The higher the concentration of syrup, the more likely crystallization is to occur. For example, it is possible for a highly concentrated syrup (eg 80° Brix) to crystallise at temperatures between -10°C-70°C. Such highly concentrated syrups must be kept at a temperature outside this range to avoid crystallization as would be understood by a skilled worker.

The supernatant liquid (or mother liquor) comprises 20% fructose, 40% glucose, 5% gluconic acid, 30% galactose and 5% others by weight of total carbohydrate and is sweeter than the composition produced by steps (i), (ii) and (iii) as galactose which has been removed, is less sweet than the remaining mixture of carbohydrates.

Thus the "mother liquor" composition is useful as a sweetener in the food industry and in particular, as it is produced from a dairy source, i.e. lactose, as a sweetener of dairy foods such as yogurt, mousse, ice cream, cream, sweetened milk drinks, etc.

The "mother liquor" is more stable than the syrup produced by the process of the first embodiment as it contains less galactose and is enriched with the "other" component and is therefore less prone to crystallisation.

The "mother liquor" may be subjected to process steps (i), (ii) and/or (iii) or any combinations thereof to further modify its composition as would be understood by a skilled worker.

The purified galactose produced by the process of the invention may be added to the composition of the invention to increase the galactose content which would provide a superior syrup for use in sports drinks or sports bars. In particular, pure galactose may be added to the compositions of the invention to increase the galactose content to a desired level.

The invention will now be exemplified.

EXAMPLES

Example 1

Lactose (BDH, 45g) was dissolved in 255g tap water. The pH of the solution was adjusted to pH 5 with citric acid. The flask was heated to 50°C in a waterbath, and lactase (Fungal Lactase, 0.90g, substrate:enzyme ratio, 80:1) was stirred in. Hydrolysis was allowed to proceed at 50°C for 24 hours. The solution was then cooled, and analysed for glucose. The glucose concentration was 7.1%.

The solution was then divided into 2 portions, A, 200g and B, 100g. Calcium carbonate (1.94g) and glucose oxidase (Fermizyme 1500, 0.1%) and catalase

(Catazyme 25L, 0.1g) were added to portion B in a flask and the flask vigorously shaken by a mechanical shaker in a water bath at 50°C for 4 hours.

Portion A was placed in a flask and heated to 60°C. Glucose isomerase (Sweetzyme IT, 2g) was added and kept in suspension by gentle shaking in a shaking incubator at 60°C. After 2 hours the Sweetzyme was allowed to sediment, and the supernatant solution was decanted from the settled enzyme through a filter paper (Whatman 541).

Both portions, A and B, were analysed for lactose, galactose and glucose, and then the solutions were mixed. The composition (w/w) of the product was 3.62% glucose, 5.58% galactose, 1.09% fructose, 0.33% oligo/di-saccharides and 0.87% gluconic acid and 11.5° Brix. This corresponded to a sugar composition, on a dry weight basis, of 31.5% glucose, 48.5% galactose, 9.5% fructose, 7.6%gluconic acid and 2.9% oligo/di-saccharides.

Example 2

Milk permeate was obtained by ultrafiltration of skim milk and had the composition: 4.6% lactose, 0.47% ash, pH 6.5. Permeate (1 kg) was placed in a flask and adjusted to pH 7.2 with magnesium carbonate (0.1g). The flask was heated to 40°C in a water bath and gently stirred. Lactase (Maxilact L2000, 1.25g, substrate:enzyme ratio, 40:1) was added and incubated at 40°C for 4 hours. The pH of the permeate was measured at intervals and maintained at 7.4 to 7.2 by additions of 1M HCl (1.25 ml total). After 4 hours an aliquot of the permeate was withdrawn for glucose analysis. The glucose content was 2.1%.

The permeate was then heated to 55°C and vigorously aerated with a stream of air. Glucose oxidase (Fermizyme GO 4000 L, 0.1 ml) and catalase (Catazyme 25L, 1.0ml) were added and the pH monitored. When the pH reached 4.5, magnesium carbonate was added to raise the pH to 5.2. The pH was then kept between 4.5 and 5.2 by continuous monitoring of the pH and additions of magnesium carbonate, until 3.41g of magnesium carbonate had been added. The airflow was stopped and the temperature of the flask raised to 60°C.

The pH of the solution was raised to 7.5 by the addition of magnesium carbonate. Glucose isomerase (Sweetzyme IT, 10g) was then added and kept in suspension by gentle stirring with an overhead stirrer and incubated for 2 hours. The solution was then cooled and the Sweetzyme allowed to settle. The supernatant solution was decanted from the settled enzyme through a filter paper (Whatman 541).

The solution was analysed for glucose, galactose, fructose, lactose and gluconic acid by HPLC. The composition (%w/w) of the solution was 0.70% glucose, 1.78% galactose, 0.47% fructose, 0.64% oligo/di-saccharides and 1.05% gluconic acid, and 4.6° Brix. This corresponded to a sugar composition, on a dry weight basis, of 15.0% glucose, 38.4% galactose, 10.1% fructose, 22.6% gluconic acid and 13.7% oligo/di-saccharides.

Example 3

Lactose (BDH, 50g) was dissolved in milk permeate (1 kg) obtained by ultrafiltration of whole milk and comprising 4.6% lactose, 0.47% ash. The pH of the solution was raised to 8.0 by the addition of dipotassium hydrogen phosphate (32g). The solution was heated to 50°C and held at this temperature for 15 minutes. It was then cooled and centrifuged.

The supernatant was adjusted to pH 7.2, and lactase (Lactozyme 3000L, 2.5g) was added. The temperature was raised to 45°C and hydrolysis allowed to proceed for 6 hours. The solution was analysed for glucose. The glucose concentration was 5.13%.

The temperature was then raised to 60°C, magnesium chloride hexahydrate (0.5 g) and glucose isomerase (Sweetzyme IT, 10g) were added and kept in suspension by gentle stirring with an overhead stirrer. Incubation was continued for 2.5 hours, and then the solution was cooled, and the Sweetzyme allowed to settle. The supernatant solution was decanted from the settled enzyme through a filter paper (Whatman 541).

The isomerised solution was heated to 50°C, and sparged with oxygen. Glucose oxidase (Enzidase GO 1500, 0.25g) and catalase (Catazyme 25L, 1.0g), together with

3.5g calcium carbonate, were then added and the enzyme reactions allowed to proceed for 7 hours. The composition (%w/w) of the product was 0.04% glucose, 4.80% galactose, 1.77% fructose, 0.94% oligo/di-saccharides and 4.86% gluconic acid, and 12.4° Brix. This corresponded to a sugar composition, on a dry weight basis, of 0.3% glucose, 38.7% galactose, 14.3% fructose, 39.2% gluconic acid and 7.7% oligo/di-saccharides.

Example 4

Wyndale refined edible lactose (200g) was dissolved in deionised water (800g) and adjusted to pH 7.2 with 0.1g tripotassium citrate, 0.03g dipotassium hydrogen phosphate and 0.12g of potassium dihydrogen phosphate. The temperature of the solution was raised to 45° in a waterbath, and Lactase (Lactozyme 3000L, 3.7g, substrate:enzyme ratio, 60:1) was added. The enzymic hydrolysis was allowed to continue for 12 hours. The pH was checked from time to time, and dipotassium hydrogen phosphate added to maintain the pH at 7.0 to 7.3. After 12 hours the glucose concentration was checked and found to be 9.7%.

The temperature of the flask was raised to 55°C and the solution was sparged with oxygen. Glucose oxidase (Enzidase GO 1500, 0.56g) was added, and the pH allowed to fall to 5.2, and then maintained at this pH by the addition of 10M sodium hydroxide. Alkali was added until 7.0% of the glucose in the solution had been converted to gluconic acid (3.6ml), and then the oxygen flow was turned off, and the pH was raised to 7.5.

The solution was heated to 60°C., and then it was allowed to percolate through a column of glucose isomerase (Sweetzyme IT) at a flow rate of 2.5 bed volumes per hour. The eluate was then evaporated in a rotary evaporator until the solids content reached 73°Brix.

The composition (%w/w) of the solution was 20.59% glucose, 34.60% galactose, 12.85% fructose, 2.41% gluconic acid and 2.62% oligo/di-saccharides, and 73° Brix.

This corresponded to a sugar composition, on a dry weight basis, of 28.2% glucose, 47.4% galactose, 17.6% fructose, 3.3% gluconic acid and 3.6% oligo/di-saccharides.

The solution was allowed to cool to room temperature (20°C). After two hours crystals started to appear. After standing for three days the crystals, which amounted to about 24% of the original sugars, were filtered off and reserved for admixture with other syrups. The composition (%w/w) of the supernatant syrup was 23.17% glucose, 25.60% galactose, 12.85% fructose, 2.41% gluconic acid and 3.28% oligo/di-saccharides, corresponding to a sugar composition, on a dry weight basis, of 34.43% glucose, 38.04% galactose, 19.09% fructose, 3.58% gluconic acid and 4.87% oligo/di-saccharides. The composition of the crystals was approximately 89% galactose, 10% glucose and minor amounts of the other sugars.

It will be appreciated that it is not intended to limit the invention to the above examples only, many variations being possible, as would readily be understood by a skilled worker, without departing from the scope of the invention.

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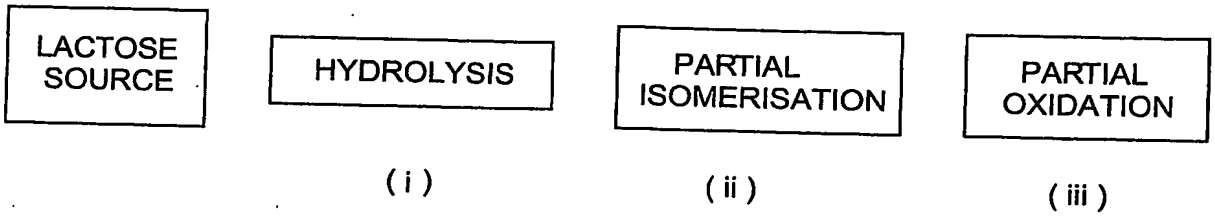

PROCESS

1. (i) → (ii) → (iii) = Composition
2. (i) → (iii) → (ii) = Composition
3. (i) + (iii) → (ii) = Composition
4. (i) → (i), (ii), (iii) = Composition
5. (i) → (ii) → (iii), (ii) = Composition
6. (i) → (iii) → (ii), (iii) = Composition

FIGURE 1